User Manual for QEDtest

HREM Research Inc.

Introduction:

The QEDtest Plug-In for DigitalMicrograph (DM) serves the purpose of testing whether a TEM is capable of acquiring large-angle rocking-beam electron diffraction (LARBED) or precession electron diffraction (PED) patterns or diffraction maps using the QED plugin. This plug-in allows the user to perform three tasks:

1. Calibrate the tilt-scan and tilt de-scan.
2. Acquire a test data stack, which consists of an on-axis diffraction pattern as well as 8 PED patterns at precession angles 1/8, 2/8, 3/8, 4/8, 5/8, 6/8, 7/8, and 8/8 times the maximum tilt angle specified. Each of the PED patterns is comprised of the sum of 6 patterns acquired for different tilts around the circle of the specified tilt magnitude.
3. Reset the illumination tilt and diffraction shift.

This Plug-In requires an online installation of DM and the capability of DM to control the illumination deflection coils as well as the diffraction shift. This functionality is commonly provided if the EMControl plug-in is installed and DM can control the TEM at all (e.g. adjust the defocus of the objective lens).

Installation:

The QEDtest Plug-In is easily installed by copying the files QEDtest.gt2 and QEDlib.gt1 to the DigitalMicrograph/PlugIns folder. Please re-start DM after copying these files.

Usage:

After successful installation the menu ‘QED’ will appear in the DM menu bar. This menu has only single item: ‘QED Test …’. Clicking on this item will open the following graphical user interface (GUI):

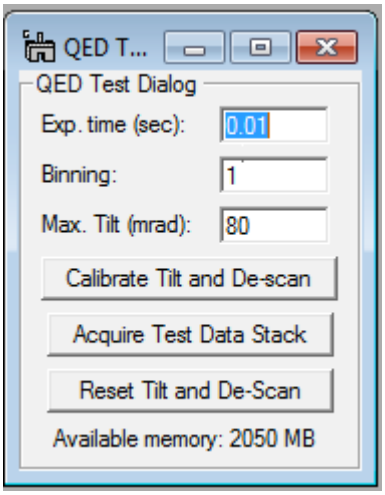

Figure 1: QED test GUI with buttons for calibration and acquisition of a test data stack.

This GUI allows the user to define the acquisition time for diffraction patterns and the desired binning, as well as the automated calibration of the tilt scan and tilt de-scan and the acquisition of the test data set.

Acquisition of the test data stack only works if the calibration has been performed for this camera length. Otherwise an error message, reminding the user of the missing calibration will appear.

Calibration:

The Calibration requires that *diffraction patterns acquired by DM are calibrated* in the units '1/µm', '1/nm', or '1/Å'. Only then can the software calibrate the illumination tilt produced by the commands that are sent to the microscope. It is suggested to have the TEM set up *in diffraction mode* but with t*he sample removed from the beam*, so that only the central spot is visible. **It is important to select the exposure time and binning such that the CCD will not be damaged**. If you are not sure how much intensity you have in your central beam, we suggest to set the exposure time to 0.01 seconds and converge your illumination. This will spread the intensity of your central beam. You can then increase the exposure time according to the dynamic range of your CCD camera. The camera length can be chosen as you would in a real experiment (e.g. about 300 mm to capture also high-order reflections.

On some installations of DM it may be necessary to make sure that DM has properly read the current microscope settings: To do this, first pressing 'Start View' in DM's own 'Camera View' floating tool box, and then pressing 'Stop View' to stop the view mode.. Please make sure that the view mode is stopped before proceeding any QEDtest or QED command.

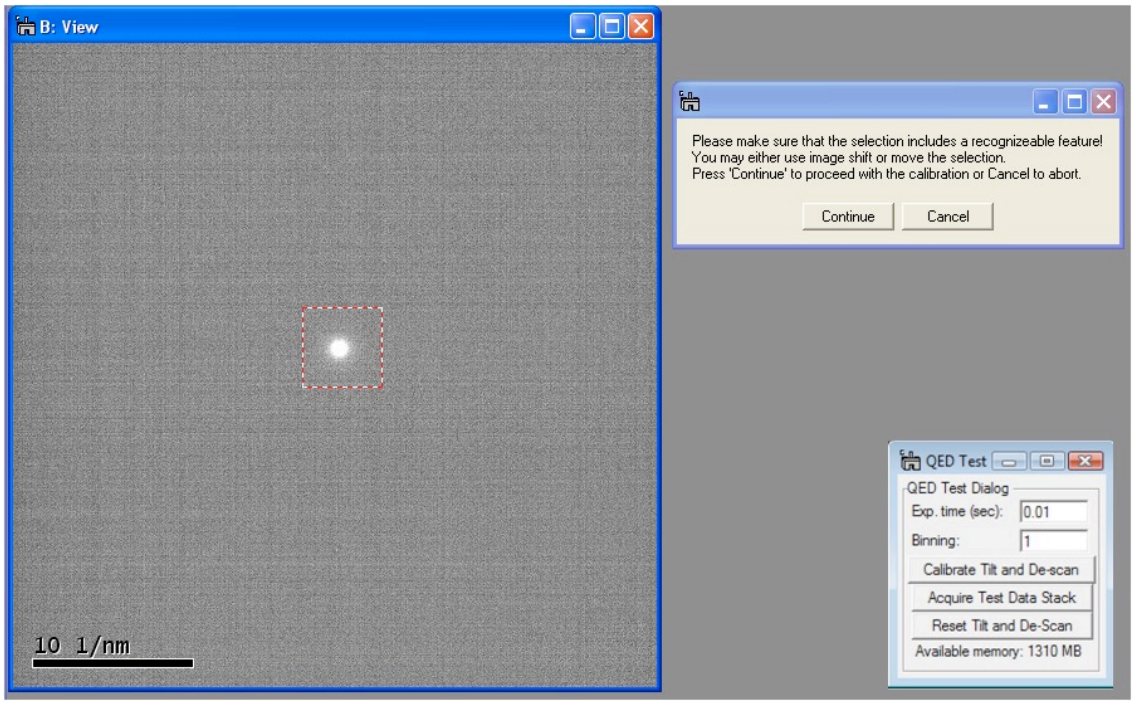

Figure 2: Screenshot demonstrating how the diffraction pattern should be set up for running the automated calibration routine.

Pressing the 'Calibrate Tilt and Descan' button will launch the fully automated calibration procedure. This procedure will first produce a continually updating image of the diffraction pattern. The user is then asked to move the central spot of the diffraction pattern well inside the region of interest (ROI) defined in the image. Alternatively, one may also move the ROI. However, one should not move the ROI too far from the image center, because otherwise the calibration routine may shift the diffraction pattern outside the area captured by the CCD.

Once the user has brought the central beam within the ROI the actual calibration may be started by pressing the 'Continue' button which should finish with the message 'Calibration successful', as shown in Fig. 3.

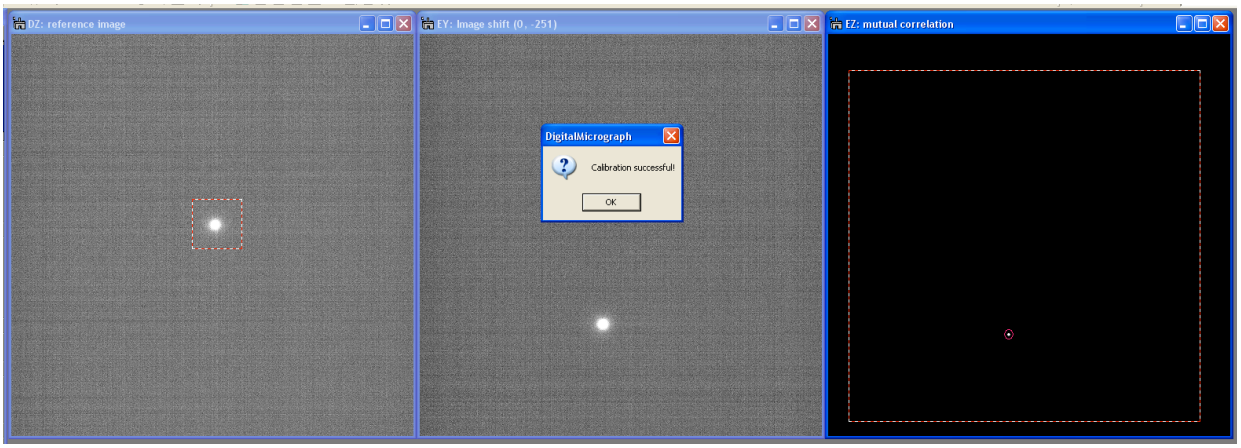

Figure 3: Screenshot of the 3 images shown during calibration. The image on the left is the reference image, the image in the center shows the tilted or shifted diffraction pattern, and the one on the far right shows a special correlation function used to measure the relative shift between the two images.

You may want to perform calibration at another camera length, say at one-half of the first calibration, and check the results. If your camera lengths are properly calibrated amplitudes of the tilt calibration should not change much with the camera length. If your diffraction pattern rotates with camera length, then the directions, but not the amplitudes, of the tilt vectors should change. If the two calibrations are almost identical, your calibration is most likely correct. The diffraction shift may or may not depend on the camera length, depending on which type of microscope you have. You may confirm the calibration of both, illumination tilt and diffraction shift, if you can get the data stack as shown in Figure 4 by using the "Acquire Test Data Stack" button.

Acquisition of the test data stack and measuring acquisition time:

Once the deflection coils have been calibrated the test data stack may be acquired. This procedure is fully automated and requires no user intervention. This function produces a data stack of nine diffraction patterns, the first one being an untilted diffraction pattern and subsequent ones being PED patterns with increasing tilt amplitude up to the maximum tilt angle you specify (set to 80 mrad by default). Each individual PED pattern is the sum of 6 diffraction patterns in which the beam is positioned at 6 different positions on the circle with

constant beam tilt amplitude. This means that a total of 1+8*6=49 individual diffraction patterns will be acquired, producing a data stack containing 9 patterns. An example data stack is shown in Fig. 4. This data set has been recorded with t*he sample removed from the beam*. This test Plug-In does not implement the aberration compensation featured by the QED Plug-In.

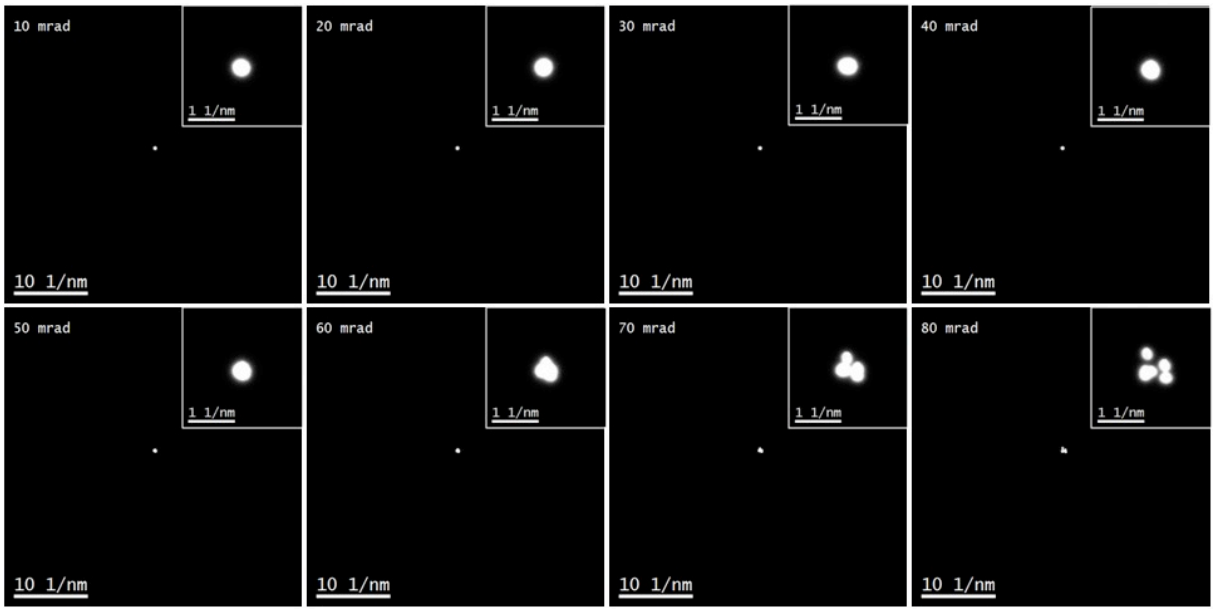

Figure 4: Example data set (only frames 1-8) recorded by the QEDtest plugin on a Zeiss EM912 without the sample being illuminated by the beam. The insets show an enlarged view of the center of the diffraction pattern. On this microscope the linear tilt de-scan calibration begins to become inaccurate at tilt amplitudes above 60 mrad (but see Fig. 5!). The first frame of the data stack, the untilted pattern is not shown.

The calibration of the tilt and tilt de-scan is linear, i.e. it is assumed that the deflection produced by the combination of commands sent to the X-tilt and Y-tilt deflectors as well as the X-diffraction-shift and Y-diffraction-shift deflectors is strictly proportional to the values submitted with the command. At large tilt angles, however, it is very likely that this linear relationship breaks down. This break-down of the linearity of the deflectors can be seen in Fig. 4 at tilt angles above 60 mrad. The QED Plug-In therefore includes a feature for correcting such alignment distortions. Fig. 5 gives an example of two PED patterns with a tilt amplitude of 120 mrad, recorded without and with the Alignment Correction.

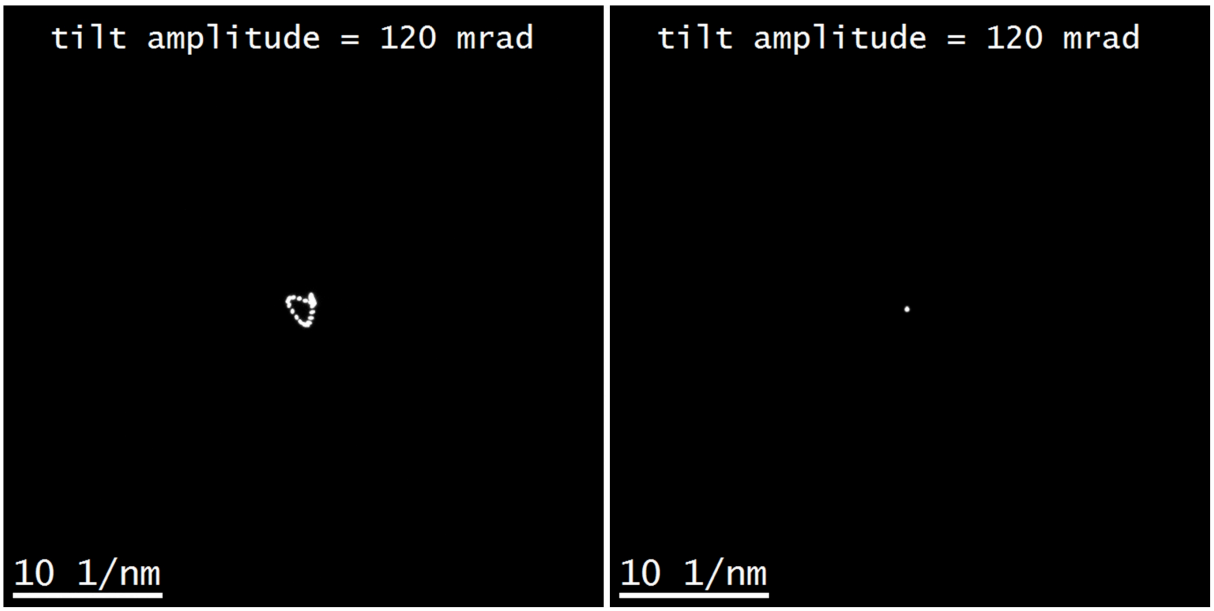

Figure 5: PED patterns recorded by the QED Plug-In on a Zeiss EM912 with a tilt amplitude of 120 mrad demonstrating the effect of the QED alignment correction. The left PED pattern has been recorded without alignment correction, and the one on the right with the ‚Alignment Correction' flag turned on.

During the acquisition of the test data stack this routine also measures the time required for different parts of the exposure and prints a report in the results window. Figure 6 shows an example output for a microscope equipped with two CCD cameras, an Orius 200W camera installed in the wide-angle port as well as a bottom-mounted MultiScan camera. This report splits the time required for each exposure into different contributions. The main components to the acquisition time of each frame are the read-out time and (for long exposures) the exposure time. Additional overhead time (named 'Other overhead time per point' in Fig. 6) is attributed to the time required to deflect the electron beam. Fig. 6 shows very clearly that the read-out time for the MultiScan CCD is much longer than for the Orius camera.

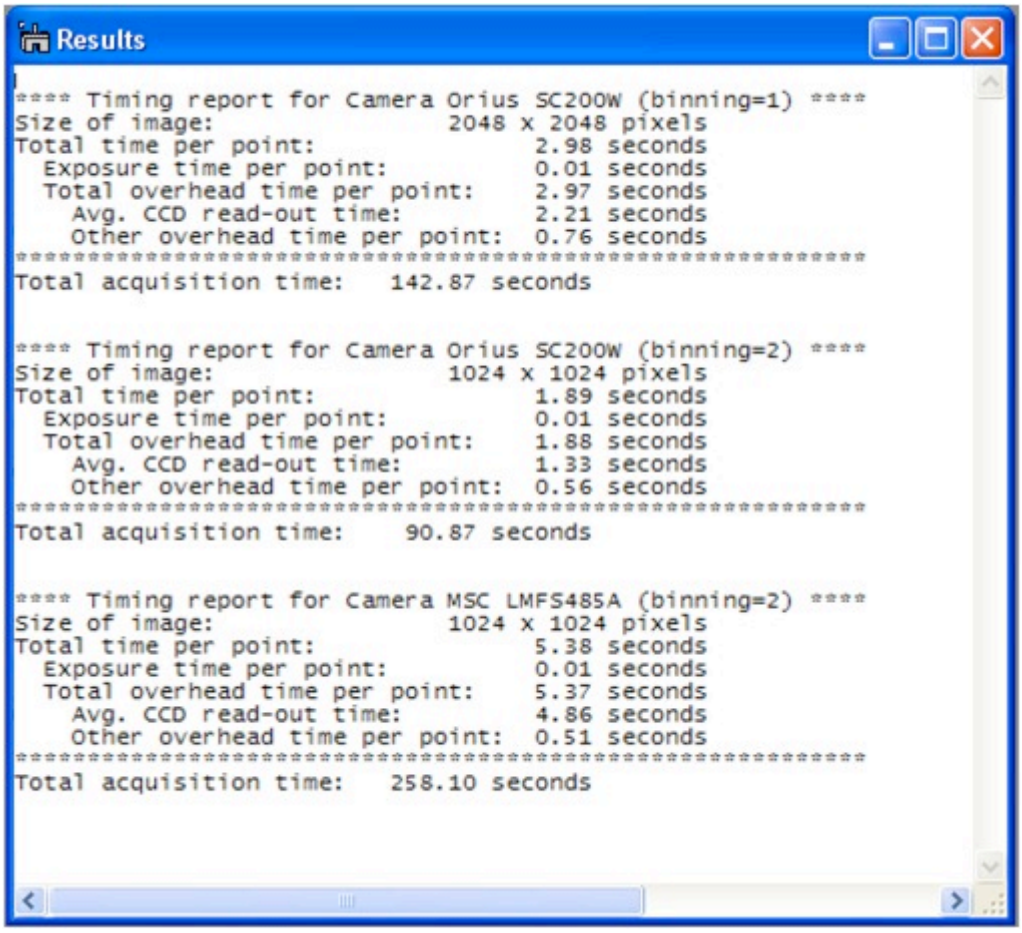
Results
**** Timing report for Camera Orius SC200W (binning=1) ****
Size of image: 2048 x 2048 pixels
Total time per point: 2.98 seconds
Exposure time per point: 0.01 seconds
Total overhead time per point: 2.97 seconds
Avg. CCD read-out time: 2.21 seconds
Other overhead time per point: 0.76 seconds
Total acquisition time: 142.87 seconds
**** Timing report for Camera Orius SC200W (binning=2) ****
Size of image: 1024 x 1024 pixels
Total time per point: 1.89 seconds
Exposure time per point: 0.01 seconds
Total overhead time per point: 1.88 seconds
Avg. CCD read-out time: 1.33 seconds
Other overhead time per point: 0.56 seconds
Total acquisition time: 90.87 seconds
**** Timing report for Camera MSC LMFS485A (binning=2) ****
Size of image: 1024 x 1024 pixels
Total time per point: 5.38 seconds
Exposure time per point: 0.01 seconds
Total overhead time per point: 5.37 seconds
Avg. CCD read-out time: 4.86 seconds
Other overhead time per point: 0.51 seconds
Total acquisition time: 258.10 seconds

Figure 6: Screenshot of the results window after acquiring test data stacks on two different cameras with two different binnings.

Reset tilt and tilt de-scan:

In the case that an unexpected error occurs during the acquisition of the test data stack (e.g. the tilt or shift limits set by the microscope manufacturer have been exceeded) the illumination tilt and diffraction shift may not be reset automatically. In this case, pressing the 'Reset tilt and de-scan' button will ensure that the illumination tilt and the diffraction shift are set again to the values they were at before the test script was started.